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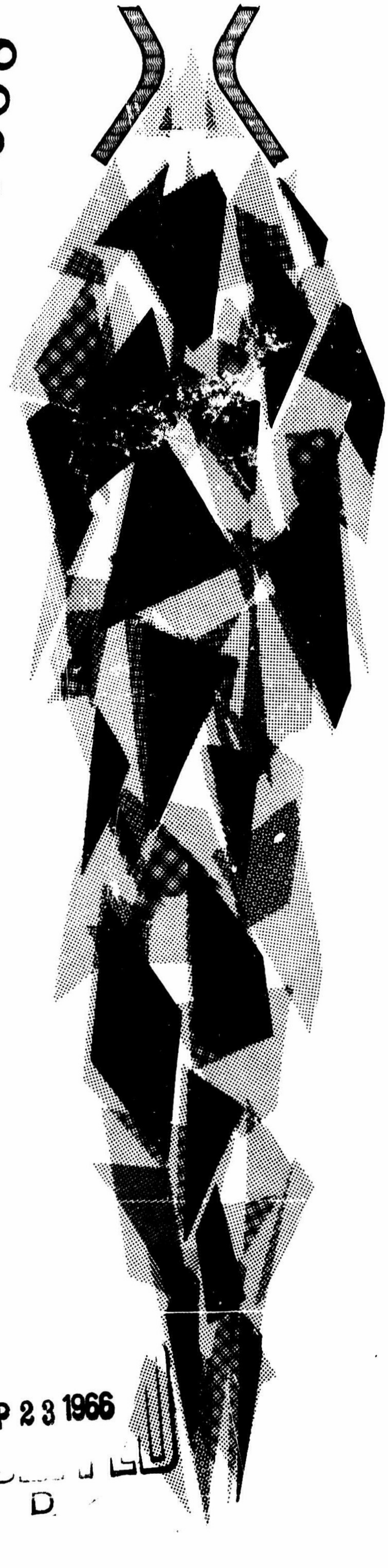

REPORT NO. S-97

SCALE-UP OF NF PREPOLYMER PROPELLANT PROCESSING

U. S. ARMY MISSILE COMMAND

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Report No. S-97

SCALE-UP OF NF PREPOLYMER PROPELLANT PROCESSING-I (U)

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SCALE-UP OF NF PREPOLYMER PROPELLANT PROCESSING-I (U)

ABSTRACT

NF prepolymer propellants were scaled up from laboratory batches (100 gms.) to pilot plant size (70 lbs.). A 1-pint completely remote facility was designed and built for the manufacture of experimental propellants on a 2-pound scale. Propellant for large motor evaluation programs was made in specially designed 1-gallon and 5-gallon facilities, using vertical planetary mixers, which eliminated personnel exposure at any time the propellant was being manipulated (mixing, casting, mandrel insertion, etc.). An 80-lbm motor was successfully cast in this manner. Future plans call for extension of this technology to a 50-gallon mixer size.

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I. INTRODUCTION

These Laboratories have for the past few years conducted research and development on propellants based on difluoramino chemicals. NF propellants were developed with high performance potential, good mechanical properties over a wide temperature range, and with formulation versatility. A Laboratories-wide Program[1] was undertaken to scale up these propellants and demonstrate them in large (20-80-lbm) motors.

During 1965, the processing of NF propellants was scaled from experimental laboratory batches (100 grams) to pilot plant size (70 pounds). To accomplish this program, techniques had to be developed to work with high energy NF propellants based on NFPA[2] and TVOPA in both 1-gallon and 5-gallon facilities. A 1-pint completely remote facility was used for experimental batches of NF propellant.

The initial work on NF propellants was based on NFPA monomer with polymerization occurring during propellant cure. The monomer propellants had low slurry viscosities, good thermal stability, and high performance, but had short pot life, high cure exotherms and excessive shrinkage. Binder systems based on prepolymers of NFPA and co-monomers were developed (1)[3] which eliminated most of the disadvantages of the monomer propellant. This report will be restricted to NF prepolymer propellant processing; the use of the term, "NF propellants," will refer to the prepolymer system.

NF propellants were processed in conventional propellant processing equipment, the relatively low slurry viscosity (1-3 kilopoise) simplifying propellant handling. Mandrel insertion after casting was used for all motor sizes. The propellant was mixed in a

[1]Contract No. DA-01-021 AMC 11608(Z).
[2]This and other acronyms are identified in the Glossary (Appendix A).
[3]Numbers in parentheses refer to references at the end of the report.

series of Baker-Perkins vertical Planetex® mixers ranging in size from 1 pint to 5 gallons; the initial phase of the scale-up program was culminated by the successful casting and firing of an 80-lbm NF motor in November, 1965.

This report describes the remote 1-pint facility and the so-called "semi-remote" 1-gallon and 5-gallon facilities developed for the manufacture of NF propellants. Processing techniques and data developed during the scale-up program are also discussed.

II. SUMMARY AND CONCLUSIONS

Initial scale-up studies on NF propellants were made in 1964 with a 1-pint vertical planetary mixer in which all operations were carried out remotely. Hazard evaluation and sensitivity studies showed that NF prepolymer propellants were readily processible and possessed no unusual hazards. Further scale-up to 1- and 5-gallon batches was accomplished during 1965, using conventional mixing equipment and remote casting technique which eliminated personnel exposure at any time at which the propellant was not at rest. Propellant batches based on NFPA-acrylic acid prepolymer as large as 70 pounds were made, and an 80-lbm motor was successfully cast and fired.

These larger scale processing studies showed that NF propellants can be handled readily using conventional propellant processing equipment. A slurry viscosity between 1 and 3 kilopoises made these propellants easily castable, and there was no pot life problem at the low (100°F) processing temperature. The remote casting process proved to be completely successful and is now used routinely on other propellant systems as well. Installation of a 50-gallon mixer is in progress for processing batches of NF propellants up to 700 pounds during the latter part of 1966.

III. NF PROPELLANT PROPERTIES

Early in 1965, a decision was made to base the Laboratories' program of NF propellant scale-up on a prepolymer of NFPA and HPMA cross-linked with HMDI. The ballistics and processing of these propellants (designated RH-SB) were extensively investigated, and facilities were designed and built for manufacturing 1-gallon batches. Composition RH-SB-103 was selected as the initial scale-up composition (Table I).

Table I

NF Propellant Formulations

	RH-SB-103 Wt%	RH-SE-103 Wt%
NFPA/HPMA[a] Prepolymer	13	--
NFPA/Acrylic Acid[a] Prepolymer	--	13
Ammonium Perchlorate	46	46
Aluminum[b]	15	15
TVOPA	26	26
Curing Agent (added)		
HMDI (diisocyanate)	0.4	--
UNOX 221 (diepoxide)	--	2.1

(a) A typical prepolymer recipe is 95/5 NFPA/comonomer by weight.
(b) Alcoa 140.

Because of thermal stability problems encountered with the NFPA-HPMA co-polymer system, a change in binder systems was made during the summer of 1965. The new binder consisted of an NFPA-acrylic acid copolymer cross-linked with diepoxide (propellant designated RH-SE, Table I). The change in binder system did not greatly affect ballistic properties, but did improve processibility and thermal stability (Table II). The NFPA-acrylic acid binder proved superior, and future propellant scale-up will be based on this system.

Table II

Some Properties of NF Propellants

(typical values)

	RH-SB-103	RH-SE-103
I° (theoretical), $\frac{lb_f\ sec.}{lb_m}$	269.5	268.8
r_{b1000} (55μ APC), in/sec	1.15	0.97
Specific Gravity	1.81	1.81
Max. Tensile Stress, psia @ 77°F	45	70
Strain at Max. Stress, % @ 77°F	20	25
Slurry Viscosity, kilopoise @ 100°F	2.5	1.5
Thermal Stability, 2" cube cracking, hours @ 80°C	20	400
Pot Life, hours @ 100°F	6	>18

IV. ONE-PINT REMOTE FACILITY

In the development and evaluation of a high energy propellant it is often not possible to obtain complete hazard evaluation data until the propellant can be scaled up to pound quantities. During 1962-1963 a completely remote facility was designed and built for casting up to 1000 grams of research propellants in motors and other test configurations which would define safety, ballistic, mechanical, and processing properties. This facility was first used for compositions based on NFPA and TVOPA.

A. Facilities

A completely remote processing facility for mixing up to 1000-gm batches and casting up to 400-gm motors without operator exposure at any time was obtained by modifying an existing bay. A rectangular window, 14-in. x 17-in., consisting of two 4-in.-thick Plexiglas® sheets separated by a 4-in. air gap, was installed in

one of the 12-in. reinforced concrete walls. The ceiling of the bay was covered with 3 feet of sand to contain shrapnel. Overhead and close-up viewing were provided by closed circuit television. Two American Machine and Foundry Company Model 8 Extended-Reach manipulators were used for all transfer operations within the bay.

The processing equipment installed in the bay (Figs. 1 and 2) consisted of the following items:

1. A 1-pint Baker-Perkins vertical planetary mixer, jacketed, stainless steel, equipped for remote dumping.
2. A 2000-gm Mettler Balance.
3. A Brookfield viscometer.
4. Two stainless steel, pneumatically-powered, vibrating solids-feeders.
5. A pneumatically-powered, vibrating casting table.
6. Three 12-in.-diameter, jacketed, steel curing ovens.
7. A hydraulic mandrel-pulling device.

FIG. 1. ONE-PINT PLANETARY MIXER IN THE REMOTE PROCESSING FACILITY

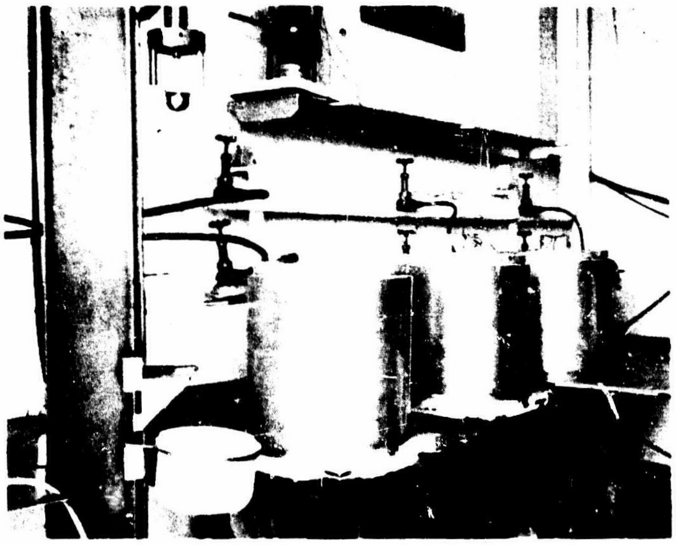

FIG. 2. CURING OVENS AND HYDRAULIC MANDREL PULLER IN REMOTE PROCESSING FACILITY

All processing operations from the weighing of liquid ingredients to transportation of the cured motors to the static firing range were conducted remotely (Fig. 3). Once processing had begun, there was no further need to enter the bay while propellant or explosive was present.

A self-propelled hazardous material carrier (HIPPO)[1] was built to bring in up to 1 pound of detonable material and to transport cured propellant to various test areas. The carrier consisted of a commercial electric warehouse truck equipped with a boiler plate personnel shield and a steel sphere to contain the explosive (Fig. 4).

The personnel shield was fabricated from 1-in.-thick steel plate on the front and 1/2-in.-thick steel plate on the sides. The

[1]This carrier is similar to one designed earlier by du Pont.

front window in the shield was made from a 1-in. thick sheet and a 4-in.-thick sheet of Plexiglas separated by a 1-in. air gap. Aluminum doors were installed on the rear of the personnel shield to reduce the blast pressure inside the cab if an explosion occurred during transfer operation.

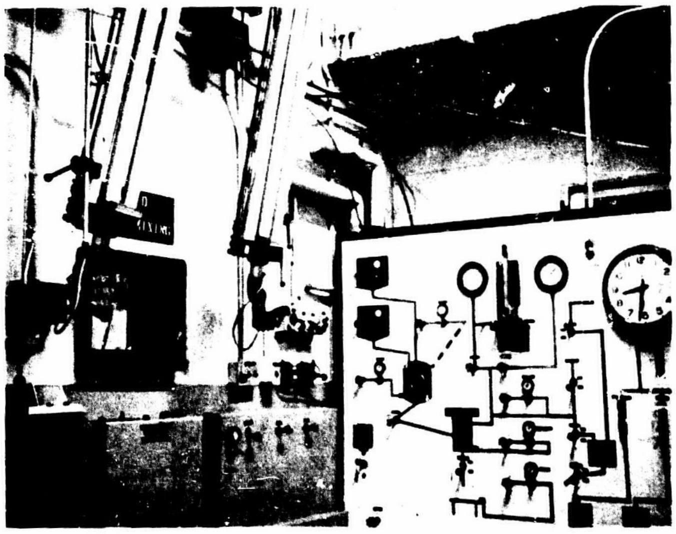

FIG. 3. CONTROL BAY FOR REMOTE PROPELLANT PROCESSING FACILITY

The steel sphere had a diameter of 30 in. and a wall 1-1/2 in. thick and contained a cup to hold the explosive. The cup and porthole cover were operated hydraulically. An American Machine and Foundry Mini-manipulator was installed in front of the personnel shield to allow transfer to and from the sphere. A stop on the manipulator kept explosives at least 20 in. from the shield.

Tests were conducted on all of the components of the HIPPO (Table III). These tests confirmed that this carrier provided adequate operator protection from the detonation of 1 pound of explosive in steel confinement.

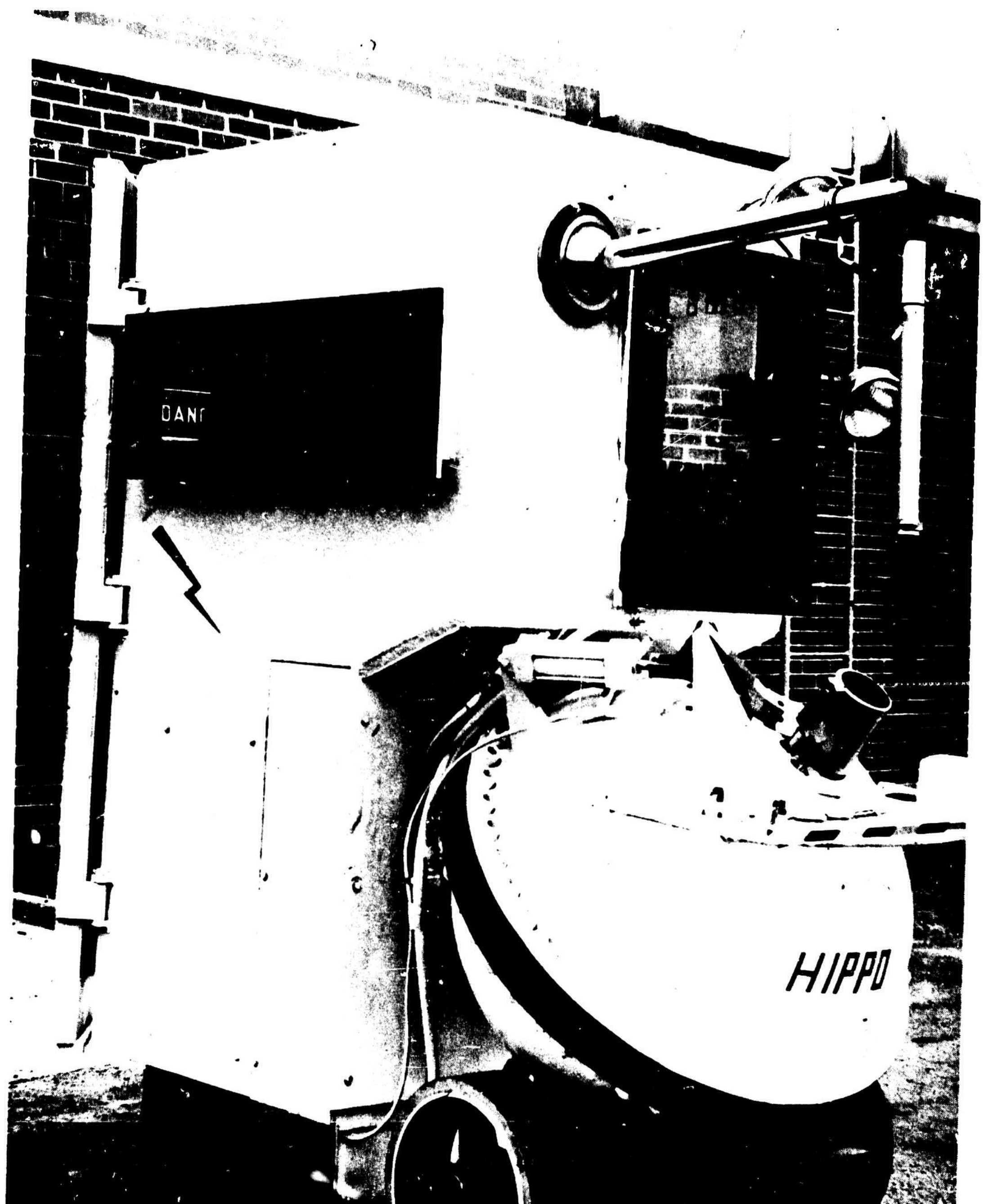

FIG. 4. SELF-PROPELLED HAZARDOUS MATERIALS CARRIER

Table III

Safaty Tests of HIPPO Components

Component Description	Tast Condition	Results
HIPPO Personnel Shiald 1 inch thick steal plata	one pound C-4 in 2-in. diametar steel pipe at 10 in.	light pimpling on oparator side; no perforation.
HIPPO Manipulator Manipulator ball joint filled with sand and mounted in 1-in. thick staal plata	331 gm. of C-4 in 2-in. diameter steel pipe at 10 in.	shrapnel panetrated exposed wall of ball joint; opposita wall undamaged.
HIPPO Window	330 gm. of C-4 in 2-in. diameter steel pipe at 10 in.	contained explosion.
Sphere	2 lb. confined C-4	puncturad 8 to 10 in. hole in bottom of sphera. Shrapnel indentation of about 3/8-in. in a 17-in. wide band around cup.[a]

[a] This was tha last of a four-shot series consisting of: 1-1/2 and 2 lb. unconfined and 1-1/2 and 2 lb. confined. No damage was noted in either of the unconfined tests and only slight damage in th 1-1/2 confined, although previous damage contributad to the damaga sustained in the 2-lbm confined test. Tha areas suffering damage were reinforced in the sphere actually usad, and tha explosive limit was set at ona pound.

B. Processing

The manufacturing process used for NFPA-TVOPA propellants in the 1-pint remote facility included the following operations:

1. Non-remote weighing of NFPA prepolymer, aluminum, ammonium perchlorate, curing agent, and cure catalyst, when applicable;
2. Non-remote charging of these ingredients to the mixer and solids hoppers:
3. Transfer of the neat plasticizer from the storage bay to the operations bay, using the HIPPO;
4. Remote weighing of the plasticizer;
5. Remote addition of the plasticizer;
6. Remote addition of aluminum while agitating;
7. Remote addition of ammonium perchlorate while agitating;
8. Mixing in the 1-pint Planetex mixer for 10 minutes;
9. Remote addition of the curing agent;
10. Vacuum mixing at about 25 mm Hg. for 10 minutes;

11. Remote measurement of slurry viscosity;
12. Remote casting and mandrel insertion;
13. Overnight curing in the bay in a nitrogen atmosphere at 140°F;
14. Remote disassembly;
15. Transfer of the cured propellant to the test area using the HIPPO.

During Steps 2-11 the mixer was heated to 100°F by circulating tempered water through the jacket.

Because of the unknown hazards associated with TVOPA, no personnel exposure to TVOPA or TVOPA-plasticized propellants was permitted during the initial phases of the evaluation program. When thermal stability and impact sensitivity of TVOPA and cured propellant containing TVOPA were found to be within the range of more familiar experimental compositions (2), the use of the HIPPO for transportation of TVOPA and cured propellant was discontinued.

During the 1-pint mixer study the process evolved from monomer propellant to RH-SB prepolymer (NFPA-HPMA) to RH-SE prepolymer (NFPA-acrylic acid). Quality control information was collected on the propellant slurries and the cured propellant. The slurry viscosity of each batch was measured with a Brookfield viscometer, a sheet mold (4 in. x 4 in. x 1/4 in.) was cast for mechanical properties, and each cured item was inspected visually and by X-ray.

The monomer process had the potentially serious disadvantages of high peak temperature and polymerization shrinkage on curing, especially in thick web motors. Following extensive laboratory investigation, it was decided early in 1965 that further, large scale evaluation would use the prepolymer process, in which cure consisted of forming a relatively few cross-links between polymer chains. Several months were spent characterizing an NFPA-HPMA (RH-SB) system cross-linked with diisocyanate. Numerous bubbles and voids were found

in the cured RH-SB propellants. These were initially attributed to an HMDI-water reaction but later investigation showed that the bubbles were air trapped during casting. They were eliminated by changing to vacuum casting with mandrel insertion. Other problems (poor thermal stability and short pot life) did persist in the RH-SB series, however. A superior system was found in NFPA-acrylic acid prepolymer (RH-SE) cross-linked with a diepoxide. This system was chosen for further scale-up work in October, 1965.

The effect of various ammonium perchlorate particle sizes on processing and mechanical properties was determined for RH-SB-103, a candidate propellant for scale-up. The slurry viscosity and cured propellant tensile strength increased with decreasing particle size, while the elongation remained virtually constant except with 8 μ oxidizer (Table IV), (Fig. 5).

Table IV

Effect of Ammonium Perchlorate Size on Viscosity and Mechanical Properties

RH-SB-103

Average Particle Diameter (microns)	Slurry Viscosity (kilopoise @ 100°F)	Tensile Strength (psi)	Elongation (%)
8	2.8	48	33
15	3.1	52	21
43	2.1	50	19
55	0.9	32	20
100	0.8	36	20
180	0.5	20	18

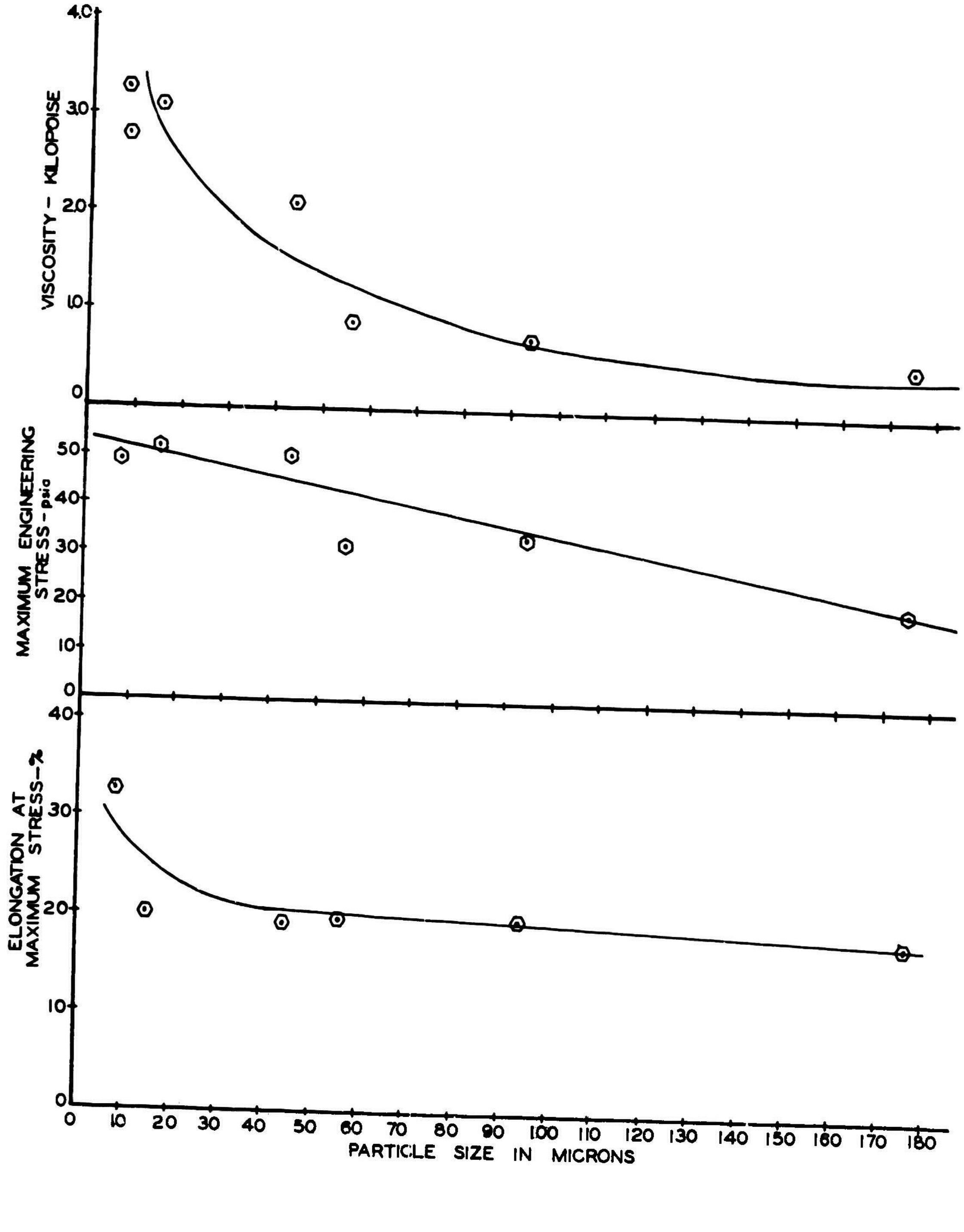

FIG. 5. EFFECT OF OXIDIZER PARTICLE SIZE ON VISCOSITY ANDMECHANICAL PROPERTIES OF RH-SB 103 PROPELLANT

All ammonium perchlorate sizes except the 8 μ were ground at these Laboratories with a hammer-mill. The small size material was purchased as-is, from American Potash and Chemical Corporation. Differences in particle shape and distribution from the different manufacturing techniques could account for the anomaly in the 8 μ data.

The oxidizer particle size had a pronounced effect on the burning rate of RH-SB-103 and on its pressure exponent above 2000 psi (Table V). The burning rate increased from 0.83 to 1.90 in./sec. at 1000 psi as the particle size was decreased from 180 to 8 μ. The pressure exponent at higher pressures increased with large particle size oxidizer.

Table V

Effect of Ammonium Perchlorate Particle Size on Burning Rate and Pressure Exponent

RH-SB-103

Average Particle Diameter (micron)	Burning Rate @ 1000 psi (in/sec)	Press. Exp. @ 1000 psi	Press. Exp. @ 2000 psi
8	1.90	0.62	0.62
15	1.65	0.56	0.56
43	1.30	0.56	0.60
55	1.15	0.56	0.66
100	0.97	0.56	0.73
180	0.83	0.60	0.75

In the entire processing history of NFPA-TVOPA propellant (400 batches, 1200 pounds propellant) only one incident occurred. During an early batch with the monomer process, an experimental mixing technique caused a fire in a small glass mixer. In this batch all of the solids (APC and aluminum) were added first, liquid was poured on top, and the mixer bowl was then raised with turbine agitator turning. The mix ignited just as the bowl was raised to mixing

position. This was attributed to frictional heat build-up from mixing partially wetted solids. No incident has occurred with the normal mixing technique in which solid materials are added gradually to the liquids.

V. SEMI-REMOTE PROCESSING

A. Processing Concept

From the propellant processing studies conducted in the 1-pint remote facility it was evident that NF propellants could be processed routinely and could be scaled up. Tests were made to determine the suitability of using a single mixer type (vertical planetary) for scaling up the NF propellants as well as for routine processing of high viscosity (PBAA) and low viscosity (plastisol double base) conventional propellants. A series of eight batches of RH-P-112 plastisol propellant (Table VI) was mixed in a 1-gallon Planetex mixer at viscosities ranging from 4,500 to 230,000 centipoise. The mechanical properties, ballistic properties, and homogeneity of each batch were determined (Table VII). Five batches of RH-P-112 propellant with the same raw materials were made in a turbine mixer as a control. This investigation showed that the planetary mixer was suitable for processing propellants over a wide viscosity range, and it was chosen for the NF propellant scale-up work.

Table VI

Composition of Plastisol Propellant RH-P-112

Ingredient	Weight %
Double Base Powder	16.67
Triethyleneglycol dinitrate	37.33
Ammonium Perchlorate	30.00
Aluminum	15.00
Resorcinol	1.00

Table VII

Comparison of Properties of RH-P 112 Made in Planetex and Turbine Mixers

	One Gallon[a] Planetex		One Gallon[b] Turbine	
	Average	Std. Deviation (Batch to Batch)	Average	Std. Deviation (Batch to Batch)
Max. Stress, psi, 77°F	55	3.5	53	2.6
Strain at Max. Stress, %, 77°F	20	1.9	20	1.3
Specific Gravity	1.651	.005	1.653	.004
Specific Impulse (I_{sps}), lbf-sec/lbm	242.0	0.44	241.8	0.71
Burning Rate (r_b), in/sec at 1000 psia	0.63	.011	0.65	.020

(a) Average of 8 batches.
(b) Average of 5 batches.

The card gap sensitivity of the NF slurry appeared to be less than those of other live binder propellants (Table VIII). Nevertheless, because of the still limited experience with the system, a means of propellant processing with minimal personnel exposure was sought. A process was designed in which all steps involving mechanical work on the propellant were conducted remotely but which permitted exposure of personnel to explosives during transportation; this was called the "semi-remote" process. A facility was built early in 1965 to cast up to 1 gallon (15 pounds) of propellant using this process. Later in the year this processing technique was extended to a nominal 5-gallon (70 pound) batch size.

Table VIII

Small-Scale Card-Gap Values of Propellant Slurries

Designation	Type	Gap, in.
RH-SB-103	NF Prepolymer Propellant	1.15
RH-P-112	Plastisol-Nitrocellulose	1.55
RH-P-197	Plastisol Nitrocellulose + RDX	1.51

B. One-Gallon Facility

The 1-gallon processing facility was built to provide NF propellant in appropriate quantities for ballistic and mechanical property scale-up studies. At the same time information could be gained from processing studies to permit the design of larger pilot plant and production facilities. The facility was designed specifically to mix up to 15 pounds of NF propellant and cast motors as large as 6-in. diameter by 18-in. long.

1) Equipment

The 1-gallon facility was housed in an existing bay, and it centered around a 1-gallon, Model 4PU Baker-Perkins vertical planetary mixer (Fig. 6). The mixer and its associated equipment were mounted on a concrete pedestal three feet above the floor (Fig. 7). A vacuum casting chamber was located next to the pedestal under a platform on which the mixer bowl could be rolled for casting (Fig. 8). Other equipment included a solvent tank for remote cleaning and a TV camera for monitoring casting. The vacuum chamber contained a 100-pound load cell for remote weighing during casting. Scales for weight-by-difference of liquid prepolymer were also located in the bay.

The control panel for operation of the facility was located approximately 25 feet away and contained controls for operating all mixing and casting steps remotely (Fig. 9). A digital readout[1] showed the weight of propellant in the motor at any time during casting. Filled motors were taken to another area for remote mandrel insertion.

Mandrels were inserted in all NF motors remotely as the final step in the casting process. An hydraulic inserter designed at Rohm and Haas supplied up to 350 pounds force to the mandrel with capacity for handling motors from 2 in. to 8 in. in diameter and mandrels up to 4 ft. in length (Fig. 10).

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[1]Type 315 digital indicator, Baldwin-Lima-Hamilton Company, Waltham, Massachusetts.

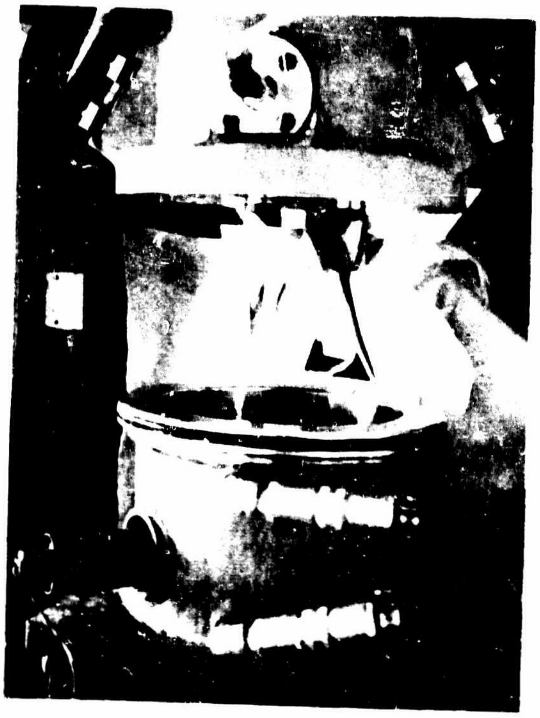


FIG. 6. ONE-GALLON VERTICAL PLANETARY MIXER

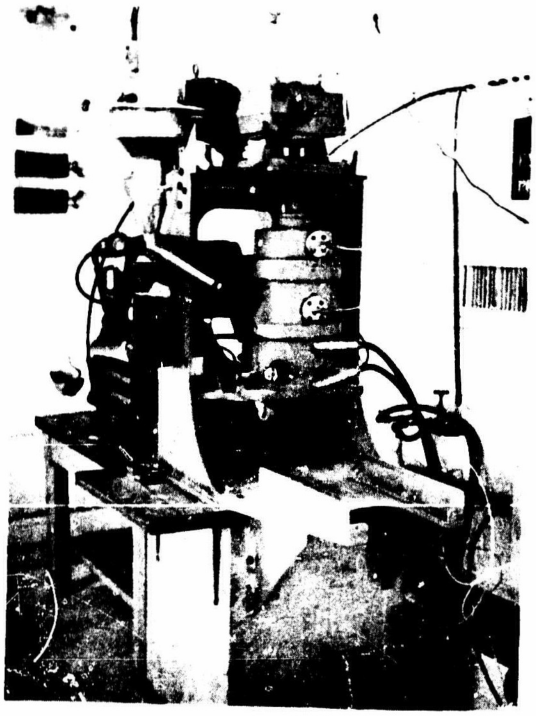


FIG. 7. ONE-GALLON MIXING FACILITY

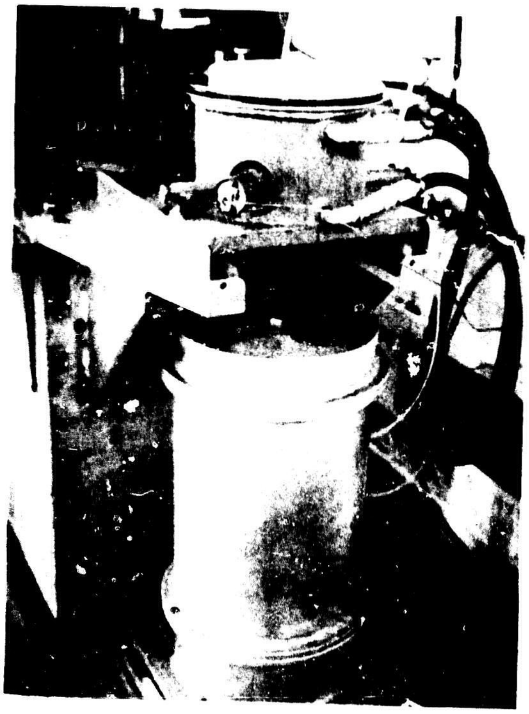


FIG. 8. ONE-GALLON MIXER IN POSITION FOR CASTING

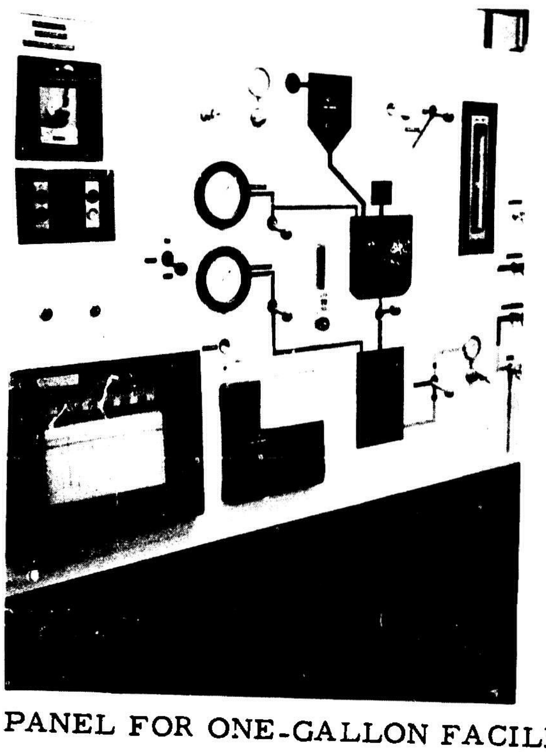


FIG. 9. CONTROL PANEL FOR ONE-GALLON FACILITY

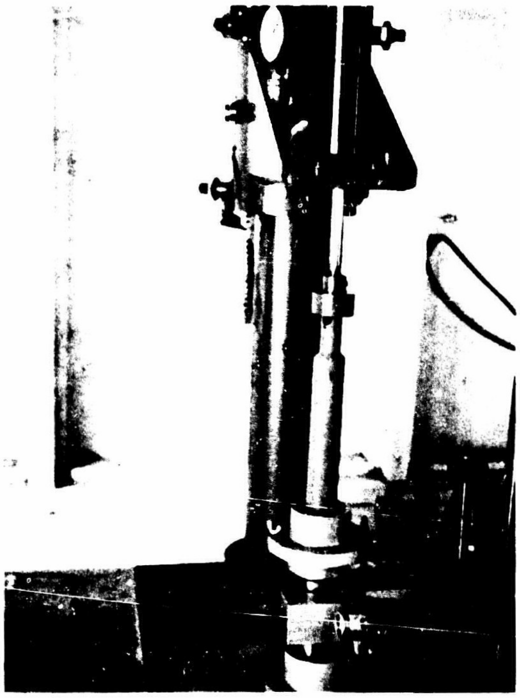


FIG. 10. REMOTE MANDREL INSERTION DEVICE

2) Processing Technique

The NF propellant semi-remote process is described in detail in Appendix B. Mixing in the 1-gallon facility was done in a conventional manner with remote addition of the oxidizer and agitation of the ingredients, vacuum being applied during the final 20 minutes of mixing. After mixing, the mixer bowl was lowered remotely, then rolled manually to a position above the casting chamber containing the motor or other item to be filled. A follower plate closely fitting the mixer bowl and made of solid Teflon®, except for a neoprene O-ring was placed on top of the mixer (Fig. 11). A vacuum line was connected to a hole in the follower, and air was withdrawn from the bowl, seating the follower against the propellant. A valve lock was removed from the bottom, a valve discharge hose attached, and the propellant was ready for casting (Fig. 8).

FIG. 11. FOLLOWER PLATE FOR ONE-GALLON MIXER

Casting was done remotely. Vacuum (29 in. of Hg. or better) was applied to the vacuum chamber, and when a pinch valve between the mixer and vacuum chamber was opened remotely, the vacuum pulled a Teflon plug from the discharge line and flow began. The amount of propellant in the motor was indicated continuously in the control room on a digital read-out from a load cell supporting the motor. When the proper weight was reached, the remote valve was closed. After release of the vacuum the bay was entered and a new motor put in place for repetition of the procedure. Mandrels were inserted remotely regardless of motor size. With this technique overfilling was a requisite, but trimming of the cured propellant was minimized by the use of a break-off fixture (Fig. 12). Clean-up was easily accomplished by feeding methylene chloride solvent remotely and under pressure through the follower plate, forcing the plate to the top of the bowl.

FIG. 12. MOTOR BREAK-OFF FIXTURE

Processing experience in this facility has been satisfactory, virtually no operating difficulties being encountered. The ease of operation and the safety of the semi-remote handling of propellant have led to the adoption of this process for all propellant processing, rather than just high energy materials. Viscosities as high as 15 kilopoises can be handled readily. Propellant yields were increased from 85 to 95% because of the small hold up in the mixer bowl and the high accuracy of the load cell weigh system, which minimized overfill in the motors.

3) Propellant Studies

Initial work with NF propellants in the 1-gallon facility was with the RH-SB series (NFPA-HPMA copolymer). With HMDI crosslinker, a relatively sluggish diisocyanate, pot life of this propellant was marginal. A series of tests was made in the 1-pint mixer to determine gel time at various temperatures. The data were reproducible and

showed that gel time was quite dependent on temperature (Fig. 13); at 90°F viscosity increased less than 50% in six hours, but at 110°F viscosity doubled in two hours. Initial viscosity data at various temperatures were also gathered (Table IX) and from these data 90°F was chosen as the processing temperature for scale-up to the 1-gallon mixer. At this temperature a compromise of reasonable viscosity and suitable pot life was obtained.

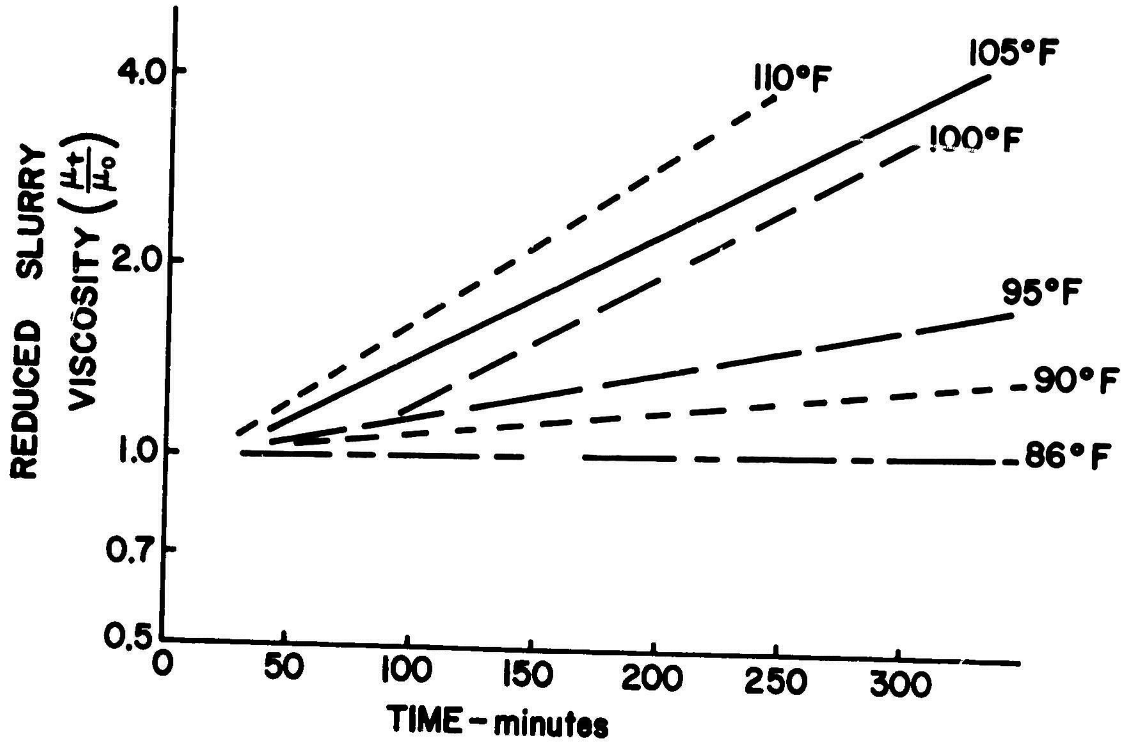


FIG. 13. SLURRY VISCOSITY VS. TIME OF RH-SB-103 PROPELLANT AT SEVERAL TEMPERATURES

Table IX

Initial Viscosity of RH-SB-103 Propellants

Slurry temperature, °F	100	90	80
Viscosity range, kilopoise	1.4 - 1.8	2.3 - 2.9	3.5 - 4.5

An increase of 30-40% in slurry viscosity resulted upon scale-up of RH-SB-103 from the 1-pint to the 1-gallon mixer with common raw materials (Table X). About the same variation in slurry viscosity with prepolymer lot was also found. Later batches of the RH-SE series propellant (NFPA-acrylic acid) did not exhibit a viscosity scale-up effect; with the latter propellant viscosity was virtually constant in 1-pint, 1-gallon, and 5-gallon batches. This viscosity increase in the RH-SB propellants was attributed to partial curing with the more reactive cross-linker as processing time increased with scale rather than a direct mixer effect. Accordingly, no trouble is expected in scaling RH-SE propellants to the 50-gallon mixer.

Table X

Scale-up Effect on Viscosity of RH-SB-103 Propellant

Mix Condition	No. Batches	Average Slurry Viscosity (kilo-poise at 100°F)	% Increase in Viscosity Over Pint Mixer
3000 centipoise binder			
1-Pt. Mixer	2	1.40	--
1-Gal. Mixer	5	1.85	32
3900 centipoise binder			
1-Pt. Mixer	2	1.90	--
1-Gal. Mixer	3	2.70	42

The change in the prepolymer (NFPA-HPMA to NFPA-acrylic acid) was made in the summer of 1965 primarily to improve thermal stability, but processibility benefitted as well because of the longer pot life and lower viscosity of the new system (Table II). Pot life problems were, in fact, eliminated with the acid prepolymer system, but it was necessary to increase the cure time at 140°F from 16 to 40 hours. Because of these demonstrated improvements composition RH-SE-

103 was chosen for scale-up to the 5-gallon mixer when an 80-lbm motor was cast in late 1965.

C. Five-Gallon Facility

The 5-gallon propellant processing facility was designed on the same principle as the "semi-remote" 1-gallon process because of the excellent processing history and proven versatility of the system. The 5-gallon facility was finished in November, 1965, and an 80-lbm motor was cast in it the same month.

1) Equipment

The mixer selected for this operation was a 5-gallon Baker-Perkins Vertical Planetex Mixer, Model 8 PI (Fig. 14), a nearly exact scale-up of the 1-gallon mixer. In contrast to the 1-gallon facility, however, bay limitations made it necessary to move the mixer bowl to a separate casting area. A 14-in.-diameter follower plate made of aluminum with a Teflon veneer was used. Double neoprene O-rings provided

FIG. 14. FIVE-GALLON VERTICAL PLANETARY MIXER

the vacuum seal (Fig. 15). A portable, hand operated lift[1] was used to raise the bowl for bottom casting into a 5-ft.-high, 6-in.-diameter vacuum chamber (Fig. 16) which held the 80-lbm motor. As before, this vessel contained a load cell which measured the propellant charged within 1%. Most other equipment was of similar design to that of the 1-gallon facility.

FIG. 15. FOLLOWER PLATE FOR 5-GALLON MIXER

[1]Portable Hoist-Model CH-5, Lewis-Shepard Company.

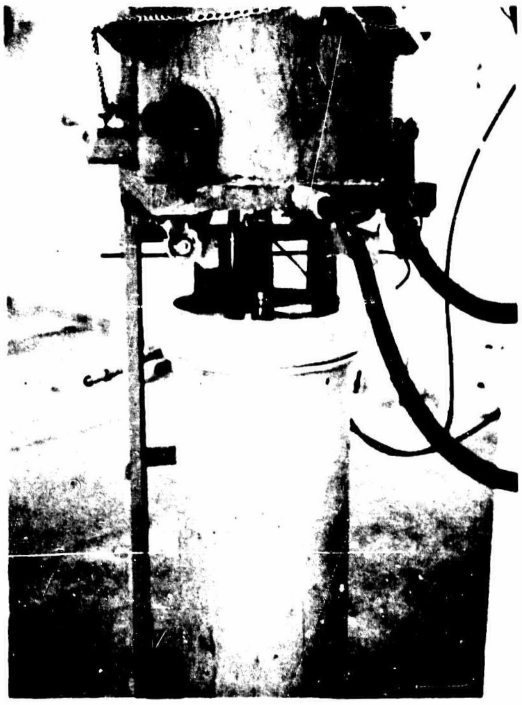


FIG. 16. FIVE-GALLON MIXER READY FOR CASTING

2) Processing

The 5-gallon semi-remote process was essentially the same as that previously described for the 1-gallon facility. Problems anticipated in the design and fabrication of a large follower plate proved not to be serious; constructed of aluminum with Teflon sheet glued to all surfaces touching the propellant or mixer bowl, it served for about 25 batches before the Teflon sheet separated. The veneer was then simply replaced, and the unit was in operation the next day. No other problem was encountered with the system; as with the 1-gallon process, propellant yields up to 95% were obtained.

3) 80-Lbm Motor

In November, 1965, an 80-lbm motor (9.1C6.5-41) was cast in the 5-gallon facility with RH-SE-103 propellant. This is, to date, the largest motor containing NF chemicals cast in any facility. A drawing of the assembled motor is shown in Fig. 17.

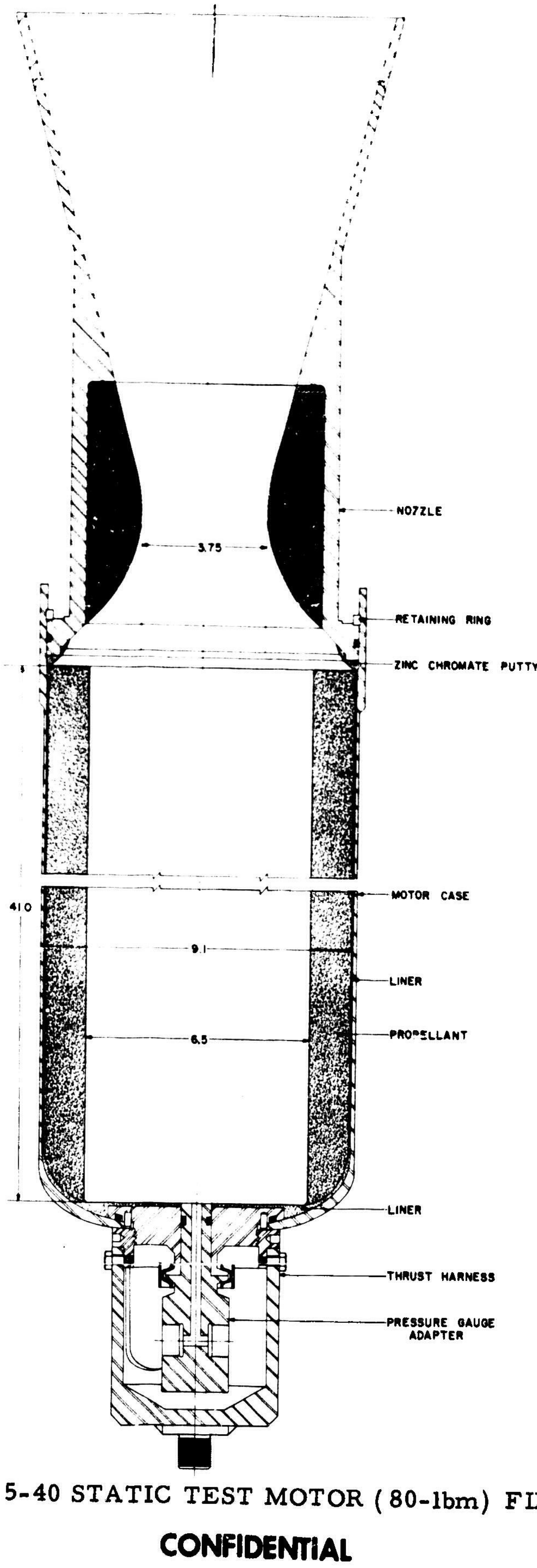


FIG. 17. 9C6.5-40 STATIC TEST MOTOR (80-lbm) FIRING ASSEMBLY

Extensive studies were made to determine optimum processing conditions for adequate pot life at a reasonably low viscosity. The processing temperature selected was 100°F. Since two batches would be required for the large motor, 2-in. motors and slabs were cast having propellant interfaces from two batches. Ballistic and mechanical properties of these were equivalent to one batch castings.

The 80-lbm motor was cast from two 50-pound batches (Table XI) and required a total mixing and casting time of eleven hours. No processing problem was encountered in casting the large motor.

Table XI

Properties of Propellant Used in 80 lbm Motor

Batch No.	Size, Gms.	Slurry Viscosity, kilopoise	Max. Stress, psi, at 77°F	Strain at Max. Stress, % at 77°F	Specific Gravity
SE-103_{cf} 1026	23,000	1.6 at 98°F	82	24	1.80
SE-103_{cf} 1027	23,000	1.6 at 97°F	83	22	1.80

After the normal 40-hour curing cycle at 140°F the casting hardware was disassembled without difficulty. Both visual and X-ray inspections showed the grain quality and the case bonding to be good.

The 80-lbm motor was successfully static tested on November 19, 1965. The specific impulse obtained was 261.2 lbf sec/lbm, which agreed well with predicted values, and the pressure time history was smooth (Fig. 18). Complete ballistic data for this firing are reported elsewhere (3).

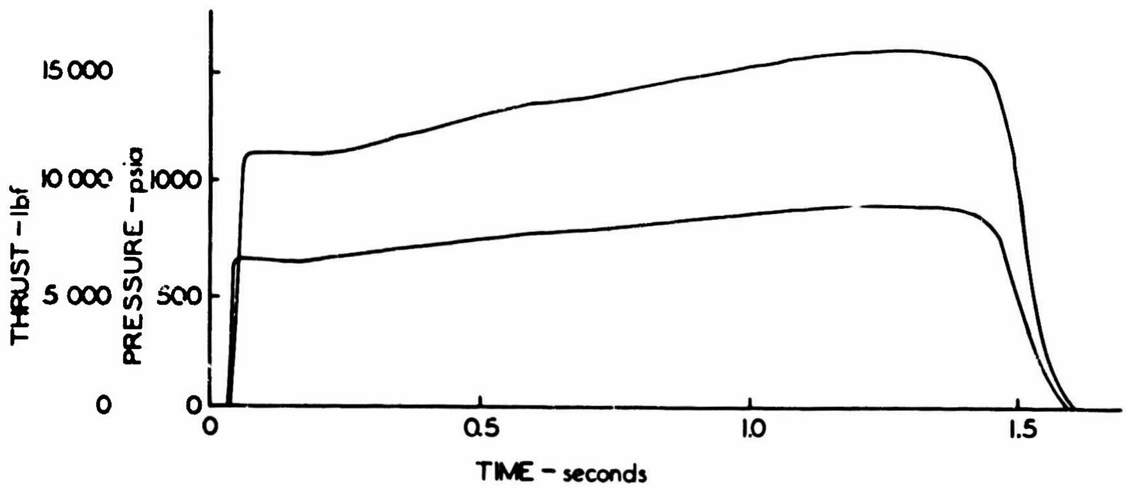


FIG. 18. PRESSURE AND THRUST TRACES FROM 80-lbm MOTOR CONTAINING RH-SE-103$_{cf}$ PROPELLANT

Since the 80-lbm motor casting, the 5-gallon facility has been used for processing up to 70-pound batches of NF propellant, primarily for casting 20-lbm (6CC18) "application" motors (Figs. 19 and 20) for characterization of the propellant system. Completely uneventful processing has been experienced in each batch; in fact, NF propellant processing in both the 1- and 5-gallon semi-remote facilities is now considered as routine a pilot plant operation as is plastisol or inert-binder composite propellant processing.

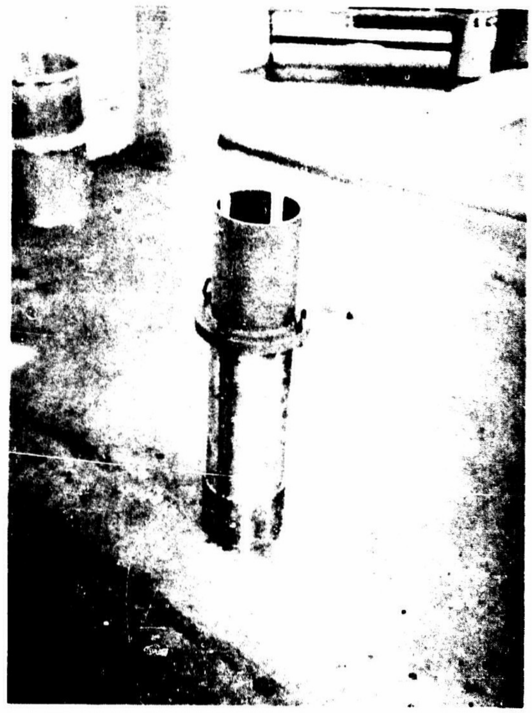


FIG. 19. 20-lbm APPLICATION MOTOR

FIG. 20. 20-lbm APPLICATION MOTOR READY FOR CASTING

D. Further Scale-Up

A mixing facility for making 50-gallon batches (70 pounds) of NF propellant is scheduled to be in operation by September, 1966. As with smaller facilities, a Baker-Perkins vertical Planetex mixer, Model No. 14PNM, will be used (Fig. 21) not only because of the previously good experience, but also because it is expected that consistent equipment will permit the development of accurate scaling predictions on propellant processibility. No major problem in processing NF propellants semi-remotely at this batch size level is anticipated although some changes in follower-plate design may be necessary because of the large (30-in.) diameter required.

With the activation of the 50-gallon mixer these Laboratories will have the capability of making NF (and other) propellants in any batch size from 100 grams to 700 pounds. Up to 2 pounds can be processed remotely, and larger batches can be made by a semi-remote process.

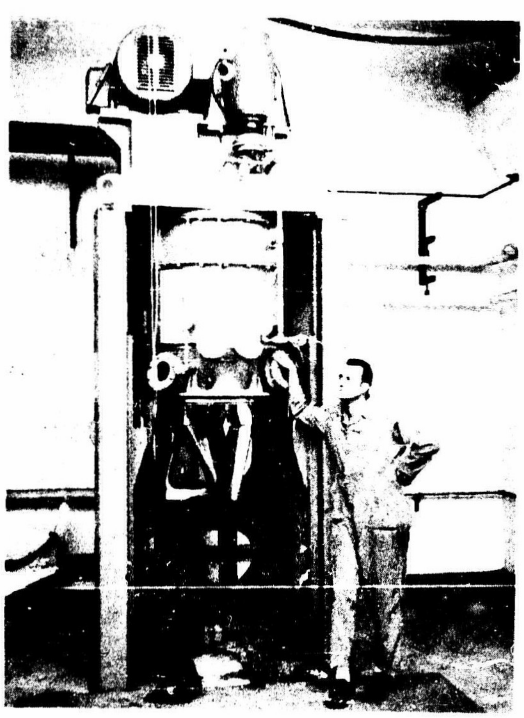


FIG. 21. 50-GALLON VERTICAL PLANETARY MIXER

REFERENCES

1. Baldwin, M. G., "Preparation and Characterization of NF Prepolymer Propellants," Rohm and Haas Company Report No. S-88, October 7, 1965.

2. Pratt, T. H., "The Explosive Characteristics of NF Compounds and Propellants," Rohm and Haas Company Report No. S-79, October 8, 1965.

3. Anderson, S. E., "Ballistic Scale-Up of NF Propellants," Rohm and Haas Company Report No. S-94, March 17, 1966.

APPENDIX A

GLOSSARY

Acronyms and Abbreviations

NFPA	2, 3-bis(difluoramino)propyl acrylate
TVOPA	1, 2, 3- tris[α, β-bis(difluoramino)ethoxy]propane
HPMA	3-hydroxypropyl methacrylate
HMDI	hexamethylene diisocyanate
UNOX 221	3, 4-epoxycyclohexylmethyl 3, 4-epoxycyclohexane-carboxylate (Union Carbide)

APPENDIX B

No. 53-19-1

Standard Operating Procedure

for

Mixing and Casting NF Prepolymer Propellants
in 1-Gallon Planetex Mixer

A. General:

The Section Safety Regulations are a part of this procedure and all applicable regulations must be observed.

B. Applicable Building and Bays: Bldg. 7593; Bays A and C

C. Safety Limits:

Bay A:	Explosives - 15 lbs.	Personnel - 3 plus supervisor
Bay C:	Explosives - 0 lbs.	Personnel - No limit

D. Check Points:

1. MIXER BAY MUST BE CLEARED OF ALL EQUIPMENT, TOOLS, etc., EXCEPT THOSE ITEMS LISTED UNDER "EQUIPMENT REQUIRED." THESE ITEMS MUST BE IN THEIR DESIGNATED STORAGE PLACES WHEN NOT IN USE.
2. ALL JEWELRY, PENCILS, KEYS, etc., MUST BE REMOVED FROM HANDS AND POCKETS OF PERSONNEL BEFORE PROCESSING IS BEGUN.
3. DURING THE OPERATION OF MIXER AND AUXILIARY EQUIPMENT, NO PERSONNEL SHALL BE IN BAY B OR ON NORTH AND WEST RAMP.
4. ALL SAFETY SIGNS MUST BE ACTIVATED BEFORE OPERATIONS ARE BEGUN.
5. VACUUM MUST BE OFF OF MIXER BEFORE THE VALVE TO LOWER MIXER IS ACTIVATED.
6. VALVES TO "LOAD CELL" HOIST MUST REMAIN IN "DOWN" POSITION EXCEPT WHEN CASTING OR ADJUSTING "READOUT."
7. ALL ITEMS PRECEDED BY ASTERISK (*) MUST BE DONE REMOTELY.

8. MIXER MUST BE INSPECTED FOR SURFACE APPEARANCE, i. e. , SCRATCHES, BULGES, INDENTIONS, etc.

9. PRECAUTIONS MUST BE TAKEN AT ALL TIMES TO MINIMIZE INHALATION OF VAPORS AND DIRECT CONTACT WITH SKIN.

E. Equipment Required:

1. Contaminated combustible scrap can
2. Non-explosive scrap can
3. Plastic spatula
4. Wooden spatula
5. Plexiglas® scraper
6. Rubber scraper
7. Feeler gauge
8. Vise grip pliers
9. Screwdriver
10. Flashlight
11. Socket wrench
12. Scale and table
13. 10-mesh screen
14. 200-gm weight
15. Solvent containers (3)
16. Stepladder
17. Cleaning brush
18. Kimwipes®

F. Personal Protective Equipment: (Not required in Control Room, Bay C)

1. Softside safety goggles
2. Flameproof coveralls or coat
3. Non-sparking shoes

G. Operating Procedure - Preliminary Operations and Checks:

1. The area must be clean and orderly before operations are begun. All tools and equipment must be accounted for and in their designated storage places.

2. The mixer and mixer blades must be dry.

3. Check mixer blade clearance each day the mixer is in operation, by using the following procedure:

Manually rotate mixer blades until the upper tip of each aligns at Point "A" (see drawing on page B-7). With feeler gauge, measure the clearance at Point "A," continuing rotation until minimum clearance is reached. Record measured clearance on data sheet.

NOTE: CONSULT SUPERVISOR IF THE MEASURED CLEARANCE IS OUTSIDE THE FOLLOWING RANGE: .100 ± .005

4. Check mixer bowl for bulges, scratches, indentions, etc., and check operation of mixer agitator. Notify supervisor of any irregularities.

5. Connect mixer bottom outlet funnel with rubber hose to vacuum chamber.

6. Install mixer valve assembly.

7. Set up vacuum chamber and check operation of load cell and "readout."

8. Check vacuum on mixer and casting can. Do not proceed until 28 inches of vacuum is reached. Record vacuum pressure on data sheet.

9. Check batch sheet for errors and initial if no error is found.

10. Turn on heating system and set temperature control at 110°F for controlling mixer temperature at 90-100°F.

11. Weigh by difference the desired amount of prepolymer-plasticizer mixture.

12. Charge all other inert ingredients except curing agent into mixer manually.

    NOTE: Aluminum powders must be screened manually through 10-mesh screen.

13. Place oxidizer container into feed hopper.

14. Push mixer into place and fasten with springs.

15. Activate safety signs on north and south entrances and ramp entrance at northwest corner of building.

16. Make sure each side of building is roped off.

17. Check tool board to be sure all items are accounted for.

18. Turn on temperature recorder.

19. Turn on load cell "readout."

20. Turn on TV camera and monitor.

21. Place mandrels in 120°F oven for preheating.
    NOTE: REPORT ANY ABNORMALITY TO SUPERVISOR.

H. Charging and Mixing: * Indicates remote operation.

*1. Raise mixer to position as indicated by pilot light.

*2. Activate "hoist" valve to "up" position and press "start" button to activate hoist.

*3. Turn mixer switch to Speed No. 2 and mix for 10 minutes. (Pilot light indicates when mixer is properly positioned.)

4. Shut down and pull stopper from oxidizer hopper.

*5. Turn on oxidizer hopper vibrator.

*6. Mix for 10 minutes.

7. Shut down and check feed hopper.

*8. Continue charging and mixing until hopper is empty.

9. When empty, check feed hopper screen for foreign objects and excessive lumps. Brush clean any hold up into mixer.

10. Prepare mixer for vacuum mixing by inserting plates over oxidizer charging ports.

*11. Turn on mixer switch to Speed No. 3 and vacuum mix for 10 minutes.

*12. BLEED VACUUM AND TURN OFF MIXER AGITATOR.

*13. Lower mixer bowl after mixer is vented.

14. Remove APC dust from mixer wall with tissue.

15. Weigh by difference the desired amount of curing agent into mixer.

*16. Raise mixer into position as indicated by pilot light.

*17. Turn on mixer switch to Speed No. 3 and vacuum mix an additional 10 minutes.

*18. BLEED VACUUM AND TURN OFF MIXER AGITATOR.

*19. Lower mixer bowl after mixer is vented. (Allow time for excess propellant to flow from mixer blades.)

20. Remove mixer bowl, place polyethylene solvent container half filled with methylene chloride under mixer blades, and energize mixer microswitch. (To be used later - I-7.)

I. Casting:

1. Place item to be cast in the casting can. (Viscosity samples should be cast first.)
2. Remove spider from mixer valve assembly.
3. Assemble vacuum chamber lid to mixer outlet.
4. Position vacuum chamber and fasten into place under lid.
5. Start Teflon follower into bowl and connect vacuum line to follower.
6. Adjust TV camera for observing Teflon follower.

*7. Raise solvent container into position. Turn mixer agitator on to Speed No. 4 to clean blades.

8. Apply vacuum to Teflon follower.

*9. Close vacuum valve when propellant rises approximately 6 inches in line from follower as indicated on TV monitor.

*10. Close casting valve in vacuum chamber.

*11. Open valve to evacuate vacuum chamber; 28 inches should be reached before proceeding.

*12. Open valve to raise load cell to support item to be filled.

*13. Zero the load cell "readout."

*14. Open casting valve in vacuum chamber. Close when desired weight is reached as indicated by load cell "readout."

*15. Release vacuum on vacuum chamber and close valve to lower load cell.

*16. Turn off mixer agitator and lower solvent container.

17. Move vacuum chamber to position under hoist. Lock in place.
18. Hoist motor from vacuum chamber and place on cart.
19. Position vacuum chamber and fasten in place under lid, and repeat items 10 through 18 until casting operation is complete.
20. Attach tube on follower to solvent line.

    NOTE: Cast all excess propellant into polyethylene container, making sure mixer is empty as indicated by load cell readout.

21. Pick up mandrels from preheating oven.

22. Transport motors to Bldg. 7597 for mandrel insertion.

*23. Insert mandrels using remote mandrel insertion device.

24. Transport motors to curing ovens.

J. Clean Up:

1. Fill solvent tank with methylene chloride for cleaning mixer bowl.

2. Adjust TV camera for observing follower.

*3. Open valve to solvent can to raise follower and close when follower reaches top of mixer as indicated by TV.

4. Remove clean-up can from under mixer blades. Place follower in can.

5. Place vacuum chamber under lid.

6. Open casting valve to vacuum chamber to empty solvent from mixer bowl.

7. Clean up any spillage.

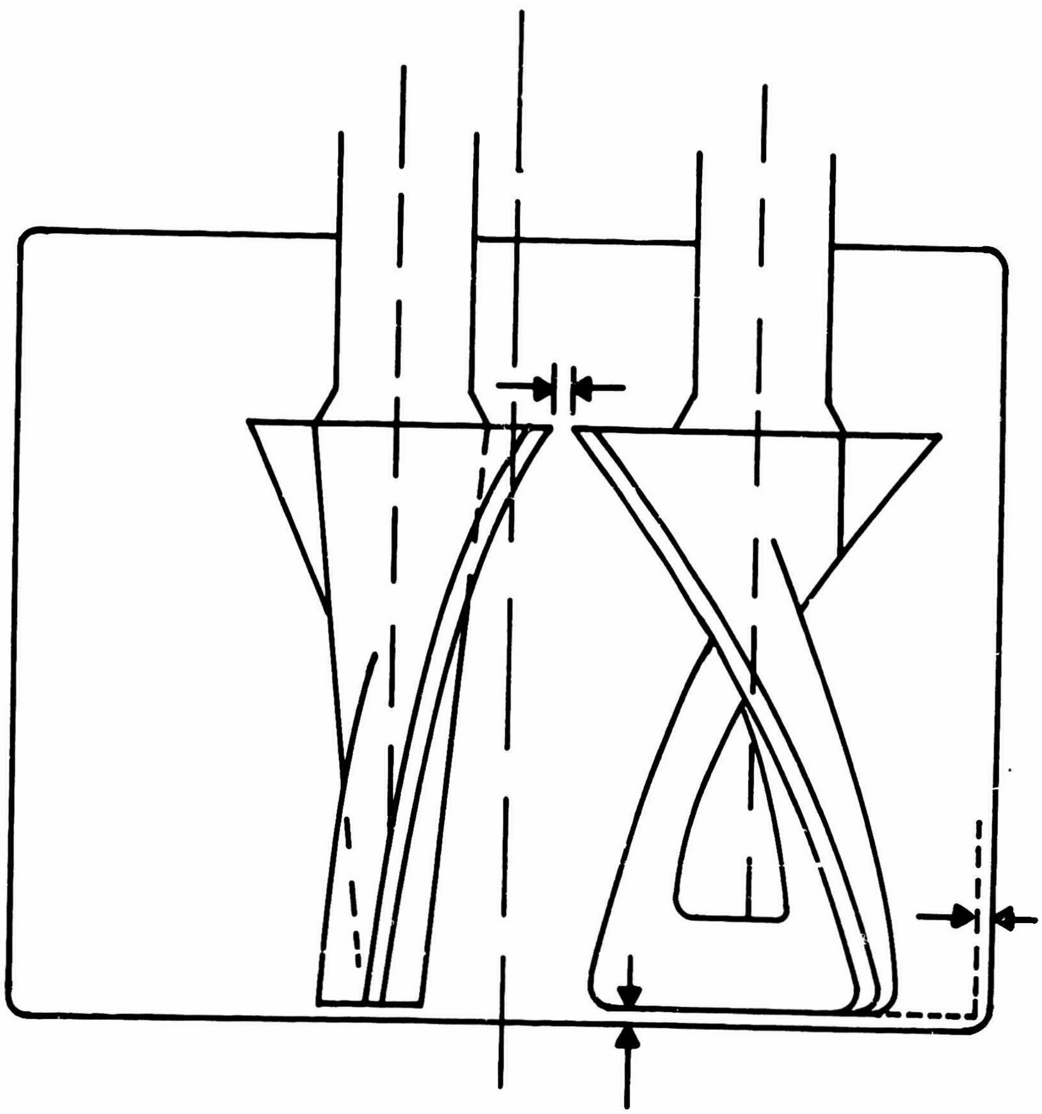
8. Remove all safety signs.

9. Turn off vacuum pump, recorder, load cell "readout," TV, etc.

10. Place all explosive scrap in a properly designated scrap container, and transport to scrap explosive storage.

NOTE: IN CASE OF MALFUNCTION OF EQUIPMENT OR CONTROLS, ALL PROCESSING WILL BE HALTED IMMEDIATELY AND SUPERVISOR WILL BE NOTIFIED. NO ACTION WILL BE TAKEN EXCEPT UNDER DIRECT SUPERVISION OF SUPERVISOR. ANY AND ALL ABNORMALITIES OBSERVED WILL BE REPORTED TO THE SUPERVISOR AND RECORDED ON THE DATA SHEET.

TO SHUT DOWN FOR GENERAL EMERGENCY, LOWER MIXER AND CIRCULATE AMBIENT WATER THROUGH MIXER JACKET.



1-GALLON PLANETARY MIXER BLADE CLEARANCE

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